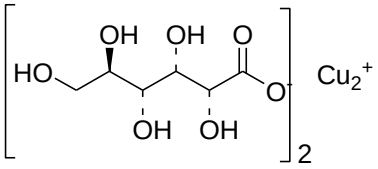
Copper Gluconate



$C_{12}H_{22}CuO_{14}$ Mol. Wt. 453.8

Copper Gluconate is copper, bis(D-gluconato-O^1,O^2).

Copper Gluconate contains not less than 98.0 per cent and not more than 102.0 per cent of $C_{12}H_{22}CuO_{14}$.

Description. A light blue to bluish-green colour powder.

Identification

A. Determine by thin-layer chromatography (2.4.17), coating the plate with silica gel G.

Mobile phase. A mixture of 50 volumes of *ethanol*, 30 volumes of *water*, 10 volumes of *ammonia* and 10 volumes of *ethyl acetate*.

Test solution. Dissolve 0.1 g of the substance under examination in *water* by heating in a water-bath at 60˚ and dilute to 10.0 ml with *water*.

Reference solution. A 1.0 per cent w/v solution of *potassium gluconate IPRS* in *water*.

Spray reagent. Dissolve 2.5 g of *ammonium molybdate* in 50 ml of *1M sulphuric acid*, add 1.0 g of *ceric sulphate* and dilute to 100 ml with *1M sulphuric acid*.

Apply to the plate 5 µl of each solution. Allow the mobile phase to raise three fourth length of the plate. Dry the plate at 110° for 20 minutes. Allow to cool, spray with spraying reagent. Dry the plate at 110° for 10 minutes. The principal spot in the chromatogram obtained with the test solution corresponds to the spot in the chromatogram obtained with the reference solution.

B. An excess of *6M ammonium hydroxide*, when added to 5 ml of 5 per cent w/v solution, produces first a bluish precipitate and then a deep blue colour solution.

Tests

Arsenic (2.3.10). Dissolve 3.3 g in 35 ml of *water*. The resulting solution complies with the limit test for arsenic (3 ppm).

Lead. Not more than 25 ppm.

Determined by atomic absorption spectrophotometry (2.4.2).

NOTE – For the preparation of all aqueous solution and for the rinsing of glassware before use, use water that has been passed through a strong acid, strong base, mixed-bed ion exchange resin. Select all reagents to have as low a content of lead as practicable, and store all reagent solutions in container of borosilicate. Cleanse glassware before use by soaking in warm 8M Nitric acid for 30 minutes and by rinsing with deionized water.

Solution A. A 1.2 per cent v/v solution of *nitric acid* in *water*.

Reference solution (a). Dissolve 159.8 mg of *lead nitrate* in 100 ml of *water*, add 1 ml of *nitric acid* and dilute to 1000 ml with *water*. Transfer 10.0 ml of the solution to a 100-ml volumetric flask, add 40 ml of *water* and 5 ml of *nitric acid* and dilute to volume with *water*.

Reference solution (b). Transfer 0.4 ml reference solution (a) to a 100-ml volumetric flask. Add 50 ml *water* and 1 ml of *nitric acid* and dilute to volume with *water*. This solution contains 0.04 ppm of lead.

Test solution (a). Dissolve 4.0 g of the substance under examination in 50 ml of *water* and 5 ml of *nitric acid*, with the aid of ultrasound and dilute to 100.0 ml with *water*. Transfer 4.0 ml of the solution to a 100.0 ml volumetric flask, add 50 ml of *water* and 1 ml of *nitric acid* and dilute to volume with *water*.

*Test solution (b).*To 10.0 ml of test solution (a), add 10.0 ml of solution A. This solution contains 0.00 ppm of added lead from reference solution (b).

*Test solution (c).*To 10.0 ml of test solution (a), add 4.0 ml of reference solution (b) and 6.0 ml of solution A. This solution contains 0.008 ppm of added lead from reference solution (b).

*Test solution (d).*To 10.0 ml of test solution (a), add 7.0 ml of reference solution (b) and 3.0 ml of solution A. This solution contains 0.014 ppm of added lead from reference solution (b).

*Test solution (e).*To10.0 ml of test solution (a), add 10.0 ml of reference solution (b). This solution contains 0.020 ppm of added lead from reference solution (b).

Chromatographic system
- Lamp: lead hallow-cathode,
- wavelength: 283.3,
Graphite tubes temperature,

temperature˚	time (s)
70	10
90	60
120	15
250 (no gas flow)	5
250	10
250 (no gas flow)	2
2000	3.2

- Argon flow rate: 3 litre per minute,
- injection volume: 20 µl.

Inject solution A, test solution (b), (c), (d) and (e). The graphite tube is temperature programmed to reach 2000˚ in about 2 minutes. When the temperature reaches 2000˚, determine the absorbance at 283.3 nm, corrected for background absorption. Inject the solution A (blank) and the test solution, determine the absorbance. Correct the absorbance value from the test solutions by subtracting from each the absorbance value from the solution (blank). Plot the corrected absorbance of the test solution versus their added lead concentrations in ppm. Intercept, determine the concentration, C, in ppm of lead in test solution (a).

Calculate the content of lead in copper gluconate using following expression

$$\text{Lead (ppm)} = \frac{C \times V}{W}$$

Where,

C - Concentration of lead in test solution (a) ppm, determined from the intercept of linear regression line,

V - Volume of solvent taken to prepare test solution (a),

W – Weight of calcium gluconate used to prepare test solution (a) (g).

Chlorides (2.3.12). 0.36 g complies with the limit test for chlorides (700 ppm).

Sulphates (2.3.17). 0.3 g complies with the limit test for sulphates (500 ppm).

Reducing substances. Not more than 1.0 per cent.

Transfer 1g of the substance under examination to a 250-ml conical flask, add 10 ml of *water* to dissolve the sample then add 25 ml of *alkaline cupric citrate*, cover the flask and boil for 5 minutes, accurately timed and cool rapidly to room temperature. Add 25 ml of *0.6M acetic acid*, 10 ml of *0.05M iodine* and 10 ml of *3M hydrochloric acid* and titrate with *0.1 M sodium thiosulphate*, using 3 ml of *starch solution*, as indicator. Carry out a blank titration.

Calculate the percentage of reducing sugar (as dextrose), using following expression.

Reducing sugar = $\frac{(V_B - V_A) \times M \times F \times 100}{W}$

Where,
V_B – Volume of 0.1M sodium thiosulphate consumed by the blank,
V_A – Volume of 0.1M sodium thiosulphate consumed by the sample,
M – Molarity of 0.1 M sodium thiosulphate,
F – Equivalency factor, 27 mg/mEq,
W – Weight of sample (mg).

Assay. Dissolve 1.5 g in 100 ml of *water*, add 2 ml of *glacial acetic acid* and 5 g of *potassium iodide*, mix and titrate with *0.1 M sodium thiosulphate* to light yellow colour. Add 2 g of *ammonium thiocyanate*, mix. Add 3 ml of *starch solution*, and continue titrating to a milk white end point. Carry out a blank titration.

1 ml of *0.1 M sodium thiosulphate* is equivalent to 0.04538 g of $C_{12}H_{22}CuO_{14}$.

Storage. Store protected from moisture.